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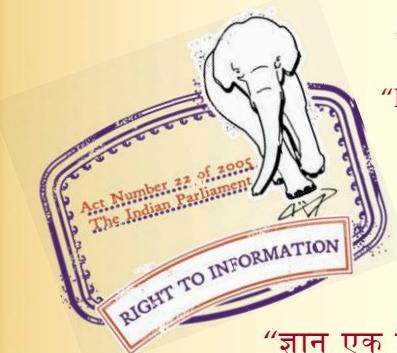
“Step Out From the Old to the New”

IS 7101 (1994): Coconut diethanolamide for cosmetic industry [PCD 19: Cosmetics]

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उद्योग के लिए – विशिष्टि

( दूसरा पुनरीक्षण )

*Indian Standard*

COCONUT DIETHANOLAMIDE FOR  
COSMETIC INDUSTRY — SPECIFICATION

( *Second Revision* )

UDC 665.58 [ 665.353.6 ]

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BUREAU OF INDIAN STANDARDS  
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

## **FOREWORD**

This Indian Standard ( Second Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

This standard was first published in 1973. First revision of this standard was done in 1983. In this second revision, changes have been made in all the requirements of Grade 2 material to align it with the current trade practices prevalent in the industry.

Coconut diethanolamide is a condensation product of fatty acids of methyl esters of coconut oil or palm kernel oil and monoethanolamine. It is used in cosmetic preparations like shampoos as a foam booster, foam stabilizer, thickening agent and wetting agent. It also possesses a mild conditioning property.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values ( revised )'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

**AMENDMENT NO. 1 SEPTEMBER 2007  
TO  
IS 7101 : 1994 COCONUT DIETHANOLAMIDE FOR  
COSMETIC INDUSTRY — SPECIFICATION**

*(Second Revision)*

[*Page 2, Table 1, col 3, Sl No. (ii)*] — Substitute ‘5.0’ for ‘9.0’.

*(Page 2, Table 1, col 4)* – Delete ‘GRADE 2’.

(PCD 19)

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Reprography Unit, BIS, New Delhi, India

## *Indian Standard*

# COCONUT DIETHANOLAMIDE FOR COSMETIC INDUSTRY — SPECIFICATION

## *( Second Revision )*

### **1 SCOPE**

This standard prescribes the requirements and methods of sampling and test for coconut diethanolamide.

### **2 GRADES**

There shall be two grades of the material, namely, Grade 1 and Grade 2.

### **3 REFERENCES**

The following Indian Standards are necessary adjuncts to this standard:

<i>IS No.</i>	<i>Title</i>
264 : 1976	Nitric acid ( <i>second revision</i> )
265 : 1987	Hydrochloric acid ( <i>second revision</i> )
266 : 1977	Sulphuric acid ( <i>second revision</i> )
323 : 1959	Rectified spirit ( <i>revised</i> )
548 (Part 1) : 1976	Methods of sampling and test for oils and fats : Part 1 Methods of sampling, physical and chemical tests
1070 : 1992	Reagent grade water — Specification ( <i>third revision</i> )
2362 : 1973	Determination of water by the Karl Fischer's method ( <i>first revision</i> )
4905 : 1968	Methods for random sampling
2088 : 1988	Methods for determination of arsenic ( <i>first revision</i> )
3958 : 1984	Methods of sampling cosmetics ( <i>first revision</i> )
4707	Classification of cosmetic raw materials and adjuncts :
(Part 1) : 1988	Dyes, colour and pigments ( <i>first revision</i> )
(Part 2) : 1993	List of raw materials generally not recognized as safe for use in cosmetics ( <i>first revision</i> )

The above mentioned standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based

on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated above.

### **4 REQUIREMENTS**

#### **4.1 Description**

Coconut diethanolamide shall be a pale straw-coloured viscous liquid.

#### **4.2 Colour and Odour**

The colour and odour of the material shall be as agreed to between the purchaser and the supplier.

**4.3** The material shall comply with the requirements given in Table 1 when tested according to the methods prescribed in col 5 of Table 1.

### **5 PACKING AND MARKING**

#### **5.1 Packing**

The material shall be packed in suitable well closed containers as agreed to between the buyer and the seller.

#### **5.2 Marking**

The containers shall be legibly marked with the following information:

- a) Name of the material;
- b) Indication of source of manufacture;
- c) Net mass of the material;
- d) Batch number in code or otherwise, to enable the lot of manufacture to be traced back from records; and
- e) Any other requirement as required by statutory authorities.

The containers may also be marked with Standard Mark.

### **6 SAMPLING**

**6.1** Representative samples of the material shall be drawn as prescribed in IS 3958 : 1984.

**6.2** Tests for all characteristics shall be carried out on composite sample.

**Table 1 Requirements for Coconut Diethanolamide for Cosmetic Industry**  
**( Clause 4.3 )**

Sl No. (1)	Characteristic (2)	Requirement		Method of Test (5)	
		GRADE 1			
		GRADE 1	GRADE 2		
i)	Free fatty acid (as lauric acid), percent by mass, <i>Max</i>	0.5	1.5	A-2	
ii)	Direct titratable amine <sup>b)</sup> (as diethanolamine), percent by mass, <i>Max</i>	9.0	18	A-3	
iii)	Combined amine <sup>b)</sup> (as diethanolamine), percent by mass, <i>Min</i>	31.0	20	A-4	
iv)	Sulphated ash, percent by mass, <i>Max</i>	1.0	1.0	A-5	
v)	Moisture, percent by mass, <i>Max</i>	1.0	1.0	IS 2362 : 1973	
vi)	Total fatty acids, percent by mass, <i>Min</i>	62.0	43.0	A-6	
vii)	Acid value of separated fatty acids	255 to 280	255 to 280	A-7	
viii)	pH of 10 percent solution	9 to 10.5	—	A-8	
ix)	Heavy metal, as lead (as Pb), ppm, <i>Max</i>	20	20	A-9	
x)	Arsenic (as As), ppm, <i>Max</i>	2	2	A-10	

<sup>b)</sup> Equivalent weight of diethanolamine to be taken as 105.

**6.3** The material shall be taken to have conformed to the specification if the composite sample passes all the tests.

**6.4** Detailed procedure of sampling and criteria of conformity of the material is given in Annex B.

## 7 TEST METHODS

Tests shall be conducted as prescribed in col 5 of Table 1. Reference to relevant standards is given in col 5 of Table 1.

## ANNEX A

*( Clauses 4.3 and 7 )*

### METHODS OF TEST FOR COCONUT DIETHANOLAMIDE

#### **A-1 QUALITY OF REAGENTS**

**A-1.1** Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1992) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

#### **A-2 DETERMINATION OF FREE FATTY ACID**

##### **A-2.0 Outline of the Method**

An alcoholic solution of the material is titrated against alkali.

#### **A-2.1 Reagents**

**A-2.1.1 Phenolphthalein Indicator** — 1 percent solution in 95 percent rectified spirit (*see* IS 323 : 1959).

**A-2.1.2 Standard Sodium Hydroxide Solution** — 0.5 N, accurately standardized.

**A-2.1.3 Neutralized Alcohol** — Add 2 ml of phenolphthalein indicator to each litre of rectified spirit and neutralize at room temperature with standard sodium hydroxide solution to a faint pink colour, just before use.

### A-2.2 Procedure

Weigh, to the nearest 0.001 g, about 10 g of the sample in a 250-ml conical flask. Without heating or melting, add 100 ml of neutralized alcohol and swirl to dissolve. Add 5 drops of phenolphthalein indicator and titrate with standard sodium hydroxide solution to the first pink colour that persists for 30 seconds.

### A-2.3 Calculation

$$\text{Free fatty acid (as lauric acid), } \frac{20.03 \times VN}{M} \text{ percent by mass}$$

where

- $V$  = volume in ml of standard sodium hydroxide solution required for titration;
- $N$  = normality of standard sodium hydroxide solution; and
- $M$  = mass in g of the sample taken for the test.

## A-3 DETERMINATION OF DIRECT TITRATABLE AMINE

### A-3.0 Outline of the Method

The material is dissolved in neutralized alcohol and titrated volumetrically with 0.5 N HCl.

### A-3.1 Reagents

**A-3.1.1 Neutralized Alcohol** — Add 2 ml of phenolphthalein indicator to each litre of rectified spirit and neutralize at room temperature with standard sodium hydroxide solution to a faint pink colour, just before use.

**A-3.1.2 Bromophenol Blue or Bromocresol Green Indicator** — Warm 0.1 g bromophenol blue or bromocresol green with 3.0 ml of 0.05 N sodium hydroxide solution and 5 ml of 90 percent ethanol. After solution is effected, add sufficient quantity of 20 percent ethanol to produce 250 ml.

**A-3.1.3 Standard Hydrochloric Acid** — 0.5 N, accurately standardized.

### A-3.2 Procedure

Weigh accurately about 4 g of the sample. Dissolve the sample completely in neutralized alcohol. Add 3 or 4 drops of the indicator and titrate with 0.5 N HCl to golden yellow colour.

### A-3.3 Calculation

$$\text{Direct titratable amine (as diethanolamine), percent} = \frac{10.5 VN}{M}$$

where

- $V$  = volume in ml of standard hydrochloric acid used in titration;
- $N$  = normality of standard hydrochloric acid; and
- $M$  = mass in g of the sample taken for the test.

## A-4 DETERMINATION OF COMBINED AMINE IN COCONUT DIETHANOLAMIDE

### A-4.0 Outline of the Method

The material is dissolved in 6 N hydrochloric acid, refluxed for 3 hours to liberate the amine which is dissolved in glacial acetic acid and titrated against 0.1 N perchloric acid potentiometrically. The total amine content is calculated, from which the value for direct titratable amine (A-3.3) is subtracted to give the combined amine content.

### A-4.1 Reagents

**A-4.1.1 Hydrochloric Acid** — 6 N.

**A-4.1.2 Rectified Spirit**

**A-4.1.3 Petroleum Ether-Range** — 40 to 60°C.

**A-4.1.4 Glacial Acetic Acid**

**A-4.1.5 Standard Perchloric Acid Reagent** — 0.1 N. To 900 ml of glacial acetic acid, add slowly with stirring 8.5 ml of 72 percent m/m perchloric acid followed by 30 ml of acetic anhydride. Allow the mixture to stand for 24 hours before use. Standardize the reagent before use. For this accurately weigh about 0.5 g of dried potassium hydrogen phthalate, dissolve in 50 ml of glacial acetic and then titrate with perchloric acid reagent, determining the end point potentiometrically.

**A-4.1.6 Crystal Violet Indicator** — 0.5 percent (m/v) solution of crystal violet in glacial acetic acid.

**A-4.1.7 Mercuric Acetate Solution** — 5.0 percent (m/v) solution of mercuric acetate in glacial acetic acid which has been neutralized to crystal violet indicator with 0.1 N perchloric acid.

### A-4.2 Procedure

Weigh accurately about 3.5 g of the sample into a 400 ml flat bottom flask. Add 50 ml of 6 N hydrochloric acid and a small magnetized stirring bar. Shake gently and mix. Connect the flask to a water-cooled reflux condenser and set on a hot plate equipped with magnetic stirring. Boil vigorously with agitation under reflux for 3 hours. Cool to room temperature. Rinse the condenser with two 5 ml portions of 1 : 1 (v/v) mixture of rectified spirit and water and add the rinsing to the flask. Transfer the contents of the flask to a 500 ml separating funnel. Rinse the flask with 50 ml of rectified spirit and add the rinsings to the separating funnel. Add 30 ml of petroleum ether and shake for 1 to 2 minutes. Remove the aqueous alcohol layer into another separating funnel.

Wash the petroleum ether layer with two 30 ml portions of 1 : 1 (v/v) mixture of rectified spirit and water. Transfer the washings to the separating funnel containing the aqueous alcohol layer.

Wash the aqueous layer with three 30 ml portions of petroleum ether and discard the washings.

Take the aqueous alcohol layer in 500 ml beaker. Heat on a water-bath to dryness. Further heat in an oven at 120°C for 30 minutes and cool in a desiccator to room temperature.

Add 100 ml of glacial acetic acid previously neutralized to crystal violet with standard perchloric acid reagent. Add 0.5 ml of crystal violet indicator. Add 60 ml of mercuric acetate solution and titrate with standard perchloric acid reagent using a magnetic stirrer, determining the end point potentiometrically.

#### A-4.3 Calculation

a) Total amine  
(as diethanolamine),  $\frac{10.51 \times VN}{M}$   
percent by mass =

where

V = volume in ml of standard perchloric acid reagent used in titration;

N = normality of standard perchloric acid reagent; and

M = mass in g of the sample taken for the test.

b) Combined amine = (total amine — direct titratable amine)

#### NOTES

1 No salt should be used at any stage of extraction, because salt interferes in potentiometric titration.

2 Although the titration is potentiometric, addition of the indicator is recommended to guide the chemist, because the indicator gradually changes its colour as the end point is approached.

### A-5 DETERMINATION OF SULPHATED ASH

#### A-5.0 Outline of the Method

The material is ignited, moistened with H<sub>2</sub>SO<sub>4</sub> and ignited again till the carbon is completely consumed.

#### A-5.1 Reagent

A-5.1.1 Concentrated Sulphuric Acid — see IS 266 : 1977

#### A-5.2 Procedure

Weigh accurately about 10 g of the sample in a suitable tared crucible. Ignite gently until the sample is thoroughly charred. Cool, moisten the residue with 1 ml of concentrated sulphuric acid and ignite gently again until the carbon is completely consumed. (Conduct the ignition in a place protected from air currents and use as low a temperature as possible to effect complete combustion of the carbon). When the carbon has completely disappeared, cool the crucible in a desiccator and weigh.

#### A-5.3 Calculation

Sulphated ash, percent by mass =  $\frac{100 M_1}{M_2}$

where

M<sub>1</sub> = mass in g of the residue, and

M<sub>2</sub> = mass in g of the sample taken for the test.

### A-6 DETERMINATION OF TOTAL FATTY ACIDS

#### A-6.0 Outline of the Method

The material is refluxed with hydrochloric acid and the fatty acids liberated are extracted with petroleum ether. The extract is then evaporated to remove the solvent and weighed.

#### A-6.1 Reagents

A-6.1.1 Hydrochloric Acid — constant boiling composition.

A-6.1.2 Methyl Orange Indicator Solution — Dissolve 0.01 g of methyl orange in 100 ml of water.

A-6.1.3 Sodium Hydroxide Solution — 50 percent.

A-6.1.4 Rectified Spirit — see IS 323 : 1959.

A-6.1.5 Petroleum Ether — range 40 to 60°C.

#### A-6.2 Procedure

A-6.2.1 Weigh 3 to 4 g of the sample to the nearest milligram directly into a small Stokes flask. Add 50 ml of hydrochloric acid and a small magnetized stirring bar. Connect the flask to a water-cooled reflux condenser and set on a hot-plate equipped with magnetic stirring. Stir and heat to boiling. Boil vigorously with agitation under reflux for 2 to 3 hours. Cool to room temperature, add 3 to 5 drops of methyl orange indicator and neutralize with sodium hydroxide solution to the disappearance of pink colour. Add 50 ml of rectified spirit and make up to the neck of the Stokes flasks with 1 : 1 (v/v) mixture of rectified spirit and water. Add 30 ml of petroleum ether and shake moderately for 1 to 2 minutes. With a siphon take off the petroleum ether layer into a tared 250 ml wide-mouthed extraction flask. Wash the aqueous alcohol layer with petroleum ether twice taking about 10 ml of petroleum ether each time and siphoning the washings into the extraction flask.

A-6.2.2 Add again about 30 ml of petroleum ether to the Stokes flask and shake vigorously for 1 and 2 minutes. Siphon the petroleum ether layer into the extraction flask. Wash the aqueous alcohol layer twice with petroleum ether as in A-6.2.1. Repeat the extraction and washing three additional times, shaking vigorously each time.

A-6.2.3 Place the extraction flask on a steam-bath and evaporate just to the last odour of petroleum ether. Gently blow into the flask and leave it on a 5 cm hole on the steam-bath for exactly 2 minutes. Cool and weigh. Replace on the 5 cm hole for exactly 1 minute, cool and weigh. The mass is considered constant when the loss in it for the heating period of 1 minute is 10 mg or less. Preserve the free fatty acids for determination of acid value as in A-7.

**A-6.3 Calculation**

$$\text{Total fatty acids, percent by mass} = \frac{100 M_1}{M_2}$$

where

$M_1$  = mass in g of the residue, and

$M_2$  = mass in g of the sample taken for the test.

**A-7 DETERMINATION OF ACID VALUE OF SEPARATED FATTY ACIDS**

**A-7.1** Determine the acid value of the fatty acids preserved as in A-6.2.3 by the method laid down in 7 of IS 548 (Part 1) : 1976.

**A-8 DETERMINATION OF pH OF 10 PERCENT SOLUTION****A-8.1 Apparatus**

A pH meter, preferably equipped with glass electrode.

**A-8.2 Procedure**

Dissolve 1 g of the sample in 10 ml of water and determine the pH of the solution with pH meter.

**A-9 DETERMINATION OF MELTING POINT****A-9.1 Apparatus**

**A-9.1.1 Oil Bath** — consisting of liquid paraffin in a glass container.

**A-9.1.2 Heat Source** — electrical or open flame, capable of being controlled to a desired rate of heating.

**A-9.1.3 Capillary Tubes** — of glass, 10 cm long and 0.8 to 1.2 mm in internal diameter.

**A-9.1.4 Thermometer** — calibrate against a certified thermometer.

**A-9.2 Procedure**

**A-9.2.1** Reduce the sample to a fine powder. Fill in a capillary tube, which is sealed at one end, sufficient quantity of dry powder to form a column 2.5 to 3.5 mm in height when packed down as closely as possible.

**A-9.2.2** Heat the bath until a temperature approximately 30°C below the expected melting point is reached. Attach capillary tube containing the powdered sample to the thermometer by wetting both with a drop of liquid paraffin and adjust its height so that the material in the capillary is in level with the thermometer bulb. Return the thermometer to the bath and continue heating with constant stirring at a rate of approximately 3 deg/min until a temperature 3°C below the expected melting point is attained. Then carefully regulate the rate to about 1 to 2 deg/min until the sample is completely melted.

**A-9.2.3** The temperature at which the column sample is observed to collapse definitely against the side of the tube at any point shall be taken as the beginning of the melting and the temperature at which the sample becomes liquid throughout shall be taken as the end of the melting.

**A-9.2.4** Both the temperature noted shall fall within the specified melting range.

**A-10 DETERMINATION OF HEAVY METALS AS LEAD (Pb)****A-10.0 Outline of the Method**

The colour produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.

**A-10.1 Apparatus**

**A-10.1.1 Nessler Cylinders** — 50 ml capacity.

**A-10.2 Reagents**

**A-10.2.1 Concentrated Hydrochloric Acid** — see IS 265 : 1976.

**A-10.2.2 Concentrated Nitric Acid** — see IS 264 : 1976.

**A-10.2.3 Dilute Acetic Acid** — 6 percent acetic acid (60 ml diluted to 1 000 ml with water).

**A-10.2.4 Hydrogen Sulphide Solution** — saturated.

**A-10.2.5 Standard Lead Solution**

Dissolve 1.600 g of lead nitrate in water and make up the solution to 1 000 ml. Pipette out 10 ml of the solution and dilute again to 1 000 ml with water. One millilitre of this solution contains 0.01 mg of lead (as Pb).

**A-10.3 Procedure**

**A-10.3.1** Place 2 g of sample accurately weighed in a platinum dish and incinerate for about 2 hours at 525 to 550°C. Cool and add 1 to 2 ml of hydrochloric acid and 0.5 ml nitric acid and evaporate to dryness on the steam bath. Dissolve the residue in 5 ml hot water, evaporate to dryness and treat it with hydrofluoric acid. Evaporate again to dryness. Dilute it with water (about 50 ml). Filter the solution, if necessary with suction through a fine fritted glass filter and dilute the filtrate and washing to 100 ml in a graduated flask. This solution shall be used for tests given in A-10.3.2 and A-11.2.

**A-10.3.2** Transfer 50 ml of sample solution prepared in A-10.3.1 in a Nessler cylinder washing it with water and add 1 ml of dilute acetic acid. In the second Nessler cylinder, place 1 ml dilute acetic acid and 2 ml of standard lead solution. Add to each cylinder 10 ml of hydrogen sulphide solution and make up the volume with water to 100 ml. Mix, allow to stand

for 10 minutes and then compare the colour produced in the two Nessler cylinders.

**A-10.3.3** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the second Nessler cylinder which is a control test.

## A-11 DETERMINATION OF ARSENIC

### A-11.0 Outline of the Method

Arsenic present in a solution of the material is reduced to arsine which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

#### A-11.1 Reagents

##### A-11.1.1 Mixed Acid

Dilute one volume of concentrated sulphuric acid with four volumes of water, and to this add 10 g of sodium chloride for each 100 ml of the solution.

##### A-11.1.2 Ferric Ammonium Sulphate Solution

Dissolve 84 g of the ferric ammonium sulphate in water containing 10 ml of mixed acid and make up to one litre.

##### A-11.1.3 Concentrated Hydrochloric Acid — see IS 265 : 1976.

##### A-11.1.4 Stannous Chloride Solution

Dissolve 80 g of stannous chloride ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ) in 100 ml of water containing 5 ml of concentrated hydrochloric acid.

## A-11.2 Procedure

Carry out the test as directed in IS 2088 : 1988, adding into the Gutzeit bottle 2 ml of ferric ammonium sulphate solution, 0.5 ml of stannous chloride solution and 25 ml of the solution as prepared in A-10.3.1. For comparison, prepare a stain using 0.001 mg of arsenic trioxide.

## ANNEX B

### ( Clause 6.4 )

## SAMPLING OF COCONUT DIETHANOLAMIDE

### B-1 GENERAL REQUIREMENTS OF SAMPLING

**B-1.0** In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

**B-1.1** The operation of drawing samples shall be carried out as expeditiously as possible.

**B-1.2** Samples shall not be taken in an exposed place.

**B-1.3** The sampling instrument shall be clean and dry.

**B-1.4** For drawing samples, the material shall not be melted.

**B-1.5** Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from external contamination.

**B-1.6** To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

**B-1.7** The samples shall be placed in clean, dry, airtight glass or other suitable material.

**B-1.8** The sample containers shall be of such a size that they are almost completely filled with the sample.

**B-1.9** Each sample container shall be sealed airtight with a suitable stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

**B-1.10** During storage, temperature of the surroundings where samples are kept shall be maintained, as far as possible, below 40°C.

### B-2 SCALE OF SAMPLING

#### B-2.1 Lot

All the containers in a single consignment of the material of the same grade drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture, the containers

belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

**B-2.1.1** Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of this specification.

**B-2.2** The number of containers ( $n$ ) to be chosen from a lot shall depend on the size of the lot ( $N$ ) and shall be as given in Table 2.

**Table 2 Number of Containers to be Selected  
( Clauses B-2.2 and B-3.1.1 )**

Lot Size ( $N$ )	Number of Containers to be Selected ( $n$ )
(1)	(2)
Up to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7
1 001 and above	8

**B-2.3** The containers shall be chosen at random. In order to ensure randomness of selection, a random number table ( see IS 4905 : 1968 ) shall be used. In the absence of such a table, however, the following procedure may be adopted:

Starting from any container in the lot, count them as 1, 2, 3, ..., up to  $r$  and so on in one order, where  $r$  is the integral part of  $N/n$ ,  $N$  being the lot size and  $n$  the sample size. Every  $r$ th container thus counted shall be drawn from the lot so as to constitute the required sample size.

### B-3 TEST SAMPLES AND REFEREE SAMPLE

#### B-3.1 Preparation of Test Samples

**B-3.1.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected ( see Table 2 ). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under 3. It shall however be not less than 250 g.

**B-3.1.2** Thoroughly mix all portions of the material drawn from a sample container. Draw equal quantity from each of the samples so obtained and mix the material collected thoroughly so as to form a composite sample weighing not less than 500 g. Divide this composite sample into three equal parts, one for the purchaser, another for the supplier and the third to be used as referee sample.

#### B-3.2 Referee Sample

The referee sample (B-3.1.2) shall bear the seals of the purchaser and the supplier and shall be kept at a place agreed to between them. It shall be used in case of dispute.

### B-4 NUMBER OF TESTS

**B-4.1** Tests for all the characteristics given under 3 shall be conducted on the composite sample.

### B-5 CRITERIA FOR CONFORMITY

**B-5.1** A lot shall be declared as conforming to this specification if the composite sample satisfies all the requirements specified under 4. If the requirement for any of the characteristics is not met, the lot shall be declared to have not satisfied the requirements of the specification.

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This Indian Standard has been developed from Doc : No. PCD 19 ( 1294 )

#### Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

## BUREAU OF INDIAN STANDARDS

### Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002  
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksansta  
( Common to all Offices )

### Regional Offices:

Telephone

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg  
NEW DELHI 110002

{ 331 01 31  
            331 13 75

Eastern : 1/14 C. I. T. Scheme VII M. V. I. P. Road, Maniktola  
CALCUTTA 700054

{ 37 84 99, 37 85 61  
            37 86 26, 37 86 62

Northern : SCO 445-446, Sector 35-C, CHANDIGARH 160036

{ 53 38 43, 53 16 40  
            53 23 84

Southern : C. I. T. Campus, IV Cross Road, MADRAS 600113

{ 235 02 16, 235 04 42  
            235 15 19, 235 23 15

Western : Manakalaya, E9 MIDC, Marol, Andheri ( East )  
BOMBAY 400093

{ 632 92 95, 632 78 58  
            632 78 91, 632 78 92

Branches : AHMADABAD, BANGALORE, BHOPAL, BHUBANESHWAR, COIMBATORE,  
FARIDABAD, GHAZIABAD, GUWAHATI, HYDERABAD, JAIPUR, KANPUR,  
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